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(54) Title: A PROCESS FOR THE PRODUCTION OF PAPER

(57) Abstract

A method for the production of paper by forming and dewatering a suspension of papermaking fibres on a The formation and dewatering take place in the presence of a cationic polymeric synthetic retention agent, preferabl tionic polyacrylamide, an anionic inorganic colloid and a polyaluminum compound. The process which is carried o stock pH above 5 gives an improved dewatering and an improved retention of fine fibres and optional fillers.

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A process for the production of paper

The present invention relates to a process for th production of paper utilizing an improved retention- an dewatering system. More particularly the invention relate to the use of a combination of a cationic polymeric reten tion agent, an anionic inorganic colloid and a polyaluminu compound as retention- and dewatering system in papermak ing.

It is previously known to use combinations of cat ionic retention agents and inorganic colloids as retention and dewatering agents in the production of paper. Th European patent application 0218674 discloses the use o polyacrylamide in combination with anionic silica sols a binders and retention agents. It is also previously know to use polymeric cationic retention agents in combinatio with polyaluminum compounds and this is disclosed in th British patent 2015614. The effect of the silica sol on fo example cationic starch with regard to retention an dewatering of the fibre web is considerably better than th effect obtained by polyaluminum compounds and cationi starch. It is assumed that one of the reasons for this i that the inorganic anionic colloids have much stronge charges than the polyaluminum compounds which have a com plex composition. It is assumed that the colloidal partic les with their strong charges produce a cross-linking o the polymeric retention agents. It is further known fro the US patent 4,643,801 to use a combination of a cationi starch, an anionic silica sol and an anionic high molecula weight polymer, particularly an anionic polyacrylamide, a a binder in papermaking. The three component system accord ing to the US patent can be used with additional aluminu compounds, such as alum, sodium aluminate or polyhydroxy aluminum chloride.

According to the present invention it has been found that the retention- and dewatering effect in papermaking is improved if a polyaluminum compound is used in combinatio with an organic, synthetic, polymeric cationic retentio agent and an anionic inorganic colloid. As the dewatering

effect is increased the speed of the papermachine can be increased and, further, less water will have to be dried off in the drying section of the paper machine.

The present invention thus relates to a process for the production of paper by forming and dewatering a suspension of papermaking fibres, and optionally fillers, on a wire whereby the forming and dewatering takes place at a pH above 5 and in the presence of an anionic inorganic colloid, a polyaluminum compound and a cationic, synthetic polymeric retention agent which is a cationic polyacrylamide or a polyethyleneimine.

The three components can be added to the fibre stock in arbitrary order. The best effect is obtained if the polyaluminum compound is added to the stock first, and then followed by addition of cationic retention agent and anionic inorganic colloid. A considerable improvement, in comparison with known technique, is obtained also when the anionic inorganic colloid is first added to the stock and the cationic polymer and the polyaluminum compound are added subsequently, in any order.

The cationic, synthetic polymeric retention agents used in the three-component system for papermaking according to the present invention are per se conventional cationic polyacrylamide and polyethyleneimine retention agents. The amount of the retention agent should be within the range of from 0.01 to 3 per cent by weight, preferably within the range of from 0.03 to 2 per cent by weight, based on dry fibres and optional fillers.

The anionic inorganic colloids which are used are also per se previously known for use in papermaking. As examples of such colloids can be mentioned colloidal montmorillonite and bentonite, titanyl sulphate sols, silica sols, aluminum modified silica sols or aluminum silicate sols. Silica based colloids are the preferred anionic inorganic colloids. The amount of anionic colloid should be within the range of from 0.005 to 2 per cent by weight, preferably within the range of from 0.01 to 0.4 per cent by weight, based on dry cellulose fibres and optional

fillers.

A preferred system which is used in combination wit a polyaluminum compound is a combination of cationi polyacrylamide and silica sol. Silica sols as disclosed i the European patent 41056, which is hereby incorporated i this application by reference, are particularly preferre and especially alkali stabilized such sols. Another pre ferred system is a cationic polyacrylamide and an anionic aluminum modified silica colloid as disclosed in th European patent application 0218674, which likewise i incorporated herein by reference.

Good results are obtained using colloidal silica i the form of an alkali stabilized sol which contains about to 60 per cent by weight of SiO_2, preferably about 4 to 3 per cent by weight of SiO_2. The colloidal silica concentration in the sol is not critical. From a practical point o view it is anyhow suitable to dilute the sols to a concentration of from 0.05 to 5.0 per cent by weight, before addition to the stock.

The colloidal silica in the sol should preferably have a specific surface of 50 to 1000 m^2/g and more preferably of about 200 to 1000 m^2/g, and the best results have been obtained when the specific surface has been about 30 to 700 m^2/g. The silica sol is stabilized with alkali in a molar ratio of $SiO_2:M_2O$ of from 10:1 to 300:1, preferably 15:1 to 100:1 (M is an ion from the group Na, K, Li and NH_4). It has been established that the colloidal silica particles should have a size below 20 nm and preferably an average particle size of from about 10 down to about 1 nm (a colloidal silica particle with a specific surface of about 550 g/m^2 corresponds to an average particle size of about 5 nm).

Silica sols which fulfil the above given specifications are available commercially, eg from Du Pont & de Nemours Corporation and Eka Nobel AB.

As has been mentioned above very good results are obtained using anionic colloidal particles which have at least a surface layer of aluminum silicate or aluminum

modified silica sol so that the surface groups of the particles contain silica and aluminum atoms in a ratio of from 9.5:0.5 to 7.5:2.5. Sols of this type also preferably have a specific surface of from 50 to 1000 m^2/g, or more preferably from 200 to 1000 m^2/g. As in the case of pure silica sols the best results have been observed at specific surfaces within the range of about 300 to 700 m^2/g.

The polyaluminum compounds which are used according to the present invention are also previously known for use in papermaking. They are termed basic and consist of polynuclear complexes. The polyaluminum compounds shall, in aqueous solution, contain at least 4 aluminum atoms per ion and preferably at least 10. The upper amount of aluminum atoms in the complexes are dependent on the composition of the aqueous phase and can vary, eg depending on the concentration and the pH. Normally the amount does not exceed 30. The molar ratio of aluminum to counter ion, with the exception of hydroxide ions, should be at least 0.4:1 and preferably at least 0.6:1.

As example of a suitable polyaluminum compound can be mentioned compounds with the net formula

$$n[Al_2(OH)_mCl_{6-m}]$$

which have a basicity of from 30 to 90%, preferably 33 to 83%. (m=2 and m=5, respectively)

Basicity is defined as the number of OH-groups divided by the number of OH groups and chloride ions x 100, ie (m:6)x100.

The polyaluminum compound can also contain anions from sulphuric acid, phosphoric acid, polyphosphoric acid, chromic acid, citric acid or oxalic acid, whereby the ratio of aluminum to such anions should be within the range of from 0.015 to 0.4

The most common type of polyaluminum compound has m=3, ie $Al_2(OH)_3Cl_3$ with a basicity of about 50%. As examples of commercially available compounds of this type can be mentioned Sachtoklar^R (sulphate free) sold by Sachtleben GmbH, F.R. Germany, WAC (contains sulphate) sold by Atochem, France, and Ekoflock (contains sulphate) sold by

Ekoflock AB, Sweden.

As another example of polyaluminum chlorides can b mentioned the highly basic polyaluminum chloride which i sold by Hoechst AG, F.R. Germany, under the name Locron an which has the net formula $[Al_2(OH)_5Cl.5H_2O]_x$ and which i aqueous solution gives the complex ion $[Al_{13}O_4(OH)_{24}(H_2O)_{12}]^{7+}$.

The amount of the polyaluminum compound can var within wide limits. It has according to the invention bee found that already very small amounts of polyaluminu compound, with regard to the amount of anionic inorgani colloid, give substantial improvements of the dewaterin effect. Improvement is obtained at a weight ratio poly aluminum compound to inorganic colloid of 0.01:1. The uppe limit is not critical. However, no improvements wort mentioning are obtained when the ratio of polyaluminu compound to inorganic colloid is greater than 3:1. Th ratio is suitably within the range from 0.02:1 to 1.5:1 preferably from 0.05:1 to 0.7:1. The ratio refers to th weight ratio between the polyaluminum compound, calculate as Al_2O_3, and the inorganic colloid.

According to the invention it is important that th pH of the stock is kept above 5, and preferably from 6 t 9. This is suitably achieved by addition of for exampl sodium hydroxide. If an alkaline filler is used, such a chalk, the suitable pH is reached without or with smalle amounts of sodium hydroxide. Other fillers than calciu carbonate can of course be used but care should be taken t keep the pH of the stock at the levels stated above.

At paper production according to the invention mine- ral fillers of conventional types can be used, eg kaolin, titanium dioxide, gypsum, chalk and talcum, can be present. The term "mineral filler" is herein used to include, besides these fillers, also wollastonite and glass fibre and also mineral low density fillers such as expande perlite. The mineral filler is usually added in the form o a water slurry in conventional concentrations used for suc fillers. Before the addition the filler can optionally be

treated with components of the dewatering- and retention system according to the invention, eg by addition of the cationic retention agent and the polyaluminum compound, or, and preferably, of the inorganic anionic colloid, whereafter the remaining component is added to the stock.

The three component system of the present invention can be used in papermaking from different types of stocks of papermaking fibres, ie stocks containing at least 50 per cent by weight of cellulosic fibres. The components can for example be used as additives to stocks from fibres from chemical pulp, such as sulphate and sulphite pulp, thermomechanical pulp, chemical thermomechanical pulp, refiner mechanical pulp or groundwood pulp, from as well hardwood as softwood. The system can of course also be used for pulps from recycled fibres.

The process according to the invention can be carried out in a known manner and with other known additions to the fibre stock, such as sizing agents etc.

The invention is further illustrated in the following examples, wherein parts and per cent relate to parts by weight and per cent by weight, unless otherwise stated.

Example 1

In the following tests the dewatering has been evaluated with a "Canadian Freeness Tester", which is the usual method for characterizing the dewatering or drainage capability according to SCAN-C 21:65.

The stock system was composed of 100% groundwood pulp with a CSF (Canadian Standard Freeness) of 110 ml. The pH of the stock was 8. The chemical additions have been calculated in kg per ton dry stock system.

The anionic inorganic colloid was an aluminum modified 15% alkali stabilized silica sol from Eka Nobel AB. The surface of the colloidal particles was modified with 9% of Al atoms and the surface area of the particles was 500 m^2/g.

The cationic polymeric retention agent was a cationic polyacrylamide, of medium cationicity, sold by Allied Colloids under the name of Percol 292.

The polyaluminum compounds used in the tests were:
- SACHTOKLAR^R from Sachtleben GmbH, F.R. Germany, with a Al_2O_3 content of 10.0%.
-WAC from Atochem, France, with an Al_2O_3 content of 10.0%
-Ekoflock from Ekoflock AB, Sweden, with an Al_2O_3 conten of 11.9%

The additions were made to 1 litre of diluted (abou 0.3%) stock with intervals of 15 seconds under agitatio (polyaluminum compound + cationic polyacrylamide + silic sol) and the flocculated stock was then passed to th freeness apparatus and measurements made 15 seconds afte the last addition. The collected water is a measure of th dewatering effect and given as ml Canadian Standard Free- ness (CSF).

The collected water was very clear after the additio of the three components and this shows that also a goo retention effect of the fines material to the fibre flock had been obtained according to the invention.

The results of the different tests with the aluminu compounds are shown in the table. The additions are calcu- lated as kg Al_2O_3 per ton dry stock, kg SiO_2 per ton dr stock, and kg polyacrylamide per ton dry stock, respective- ly.

Test No.	Polyaluminum compound kg/t	Polyacryl-amide kg/t	Colloid kg/t	CSF ml
	WAC			
1	-	-	-	110
2	-	1	-	220
3	-	1.2	-	225
4	-	2	-	235
5	-	1	2.0	320
6	-	1.2	2.0	330
7	-	1.0	2.2	340
8	-	2	2	355
9	0.2	-	-	120
10	0.2	1	-	240

11	0.1	1	2	395
12	0.2	1	2	430
13	0.3	1	2	430
14	0.4	1	2	400
15	0.1	1	1.9	390
16	0.2	1	1.8	415
17	0.3	1	1.7	420
18	0.4	1	1.6	380
Sachtoklar				
19	0.2	1	2	370
20	0.2	1	1.8	370
Ekoflock				
21	0.2	1	1.8	385

From the results shown in the table it can be seen that a combination of 2 kg/t of the silica based colloid and 1 kg/t of the polyacrylamide gives 320 ml CSF. An increase in the system in the amount of polyacrylamide from 1 to 1.2 kg gives an increase of 10 ml. An increase of the colloid from 2 to 2.2 kg gives an increase of 20 ml. An addition of only 0.2 kg of the polyaluminum compound WAC to the system of 2 kg/t of colloid and 1 kg polyacrylamide gives a CSF increase of 110 ml (from 320 to 430), while an increase of the amount of polyacrylamide from 1 to 2 kg in the system of colloid and cationic retention agent only gives an increase of 35 ml (from 320 to 355), and here it can be mentioned also that the cationic polyacrylamide is about 10 times as expensive as the polyaluminum compound.

Comparison

Comparisons were made with the same stock as above, using the same conditions, the same anionic sol and the same method of evaluation, both with systems containing cationic starch instead of the cationic polyacrylamide and with such a system including addition of an anionic polyacrylamide of medium high anionicity (PAM^-) as according to the US patent 4643801, using the order of addition as disclosed in Example III in the patent. The polyaluminum compound was the above defined WAC. The results are shown in the Table below.

Test No.	Polyaluminum compound kg/t	Cationic starch kg/t	PAM kg/t	Colloid kg/t	CSF ml
1	-	-	-	-	110
2	-	8.2	-	-	240
3	-	8.2	-	0.36	245
4	-	8.2	0.9	0.36	145
5	0.2	8.2	-	0.36	260
6	0.2	8.2	0.9	0.36	260
7	0.2	11.3	1.36	0.20	235

The results clearly show the advantages of using the present method wherein the cationic retention agent is a cationic synthetic polymeric agent and in using this in combination with an anionic inorganic colloid and a polyaluminum compound for improving drainage in papermaking.

Example 2

In this example the dewatering effect was evaluated in the same manner as in Example 1. The stock system was composed of a recycled fibres (Inland Waste pulp) with a CSF of 138 ml and the pH of the stock was 6.5.

Two different kinds of anionic silica based colloids were used. Colloid 1) was a 15% alkali stabilized silica sol with a specific surface of about 500 m^2/g (according to EP 0041056) from Eka Nobel AB. Colloid 2) was a colloidal bentonite with a specific surface in water of about 400 to 800 m^2/g. The polyaluminum compound was WAC as used in Example 1 and as cationic polymeric retention agents both the polyacrylamide, PAM, as in Example 1 and a polyethyleneimine, PEI, sold by BASF AG under the name of Polymin SK.

Also in these tests the collected water was very clear after the addition of the three components which shows that a good retention of the fibre flocks was obtained.

Test No.	Polyaluminum compound kg/t	Cationic polymer kg/t	Colloid No.) kg/t	CSF ml
	-	-	-	138
1	-	PAM 1	-	210
2	-	1	1) 2.0	260
3	0.2	1	1.8	300
4	0.4	1	1.6	320
5	1.0	1	1.0	300
6	0.2	1	-	260
7	-	1	2) 2.0	290
8	0.2	1	1.8	325
9	0.4	1	1.6	340
10	0.8	1	1.2	305
11	0.4	1	0.8	350
12	-	PEI 0.75	-	150
13	-	0.75	2) 2.0	230
14	0.2	0.75	2.0	300
15	0.3	0.75	2.0	300

Claims

1. A process for the production of paper by formin and dewatering a suspension of papermaking fibres, an optional fillers, on a wire, characterized in that th forming and dewatering takes place at a pH above 5 and i the presence of an anionic inorganic colloid, a polyal uminum compound and a cationic, synthetic, polymeri retention agent which is a cationic polyacrylamide o polyethyleneimine.

2. A process according to claim 1, characterized i that the polyaluminum compound is added to the suspensio of fibres before the cationic retention agent and th anionic inorganic colloid.

3. A process according to claim 1, characterized i that the cationic retention agent is cationic polyacryl amide.

4. A process according to claim 1 characterized i that the anionic inorganic colloid is a silica base colloid.

5. A process according to claim 4, characterized i that the colloid is a silica sol, a silica sol witl particles which have at least a surface layer of aluminu silicate or an aluminum modified silica sol.

6. A process according to claim 1, characterized i that the polyaluminum compound is a polyaluminum chloride or a polyaluminum chloride containing sulphate.

7. A process according to claim 1 or 6, characterized in that the polyaluminum compound has the net formula

$$n[Al_2(OH)_mCl_{6-m}]$$

wherein n is >4 and which has a basicity of from 30 to 90%.

8. A process according to claim 1 or 3, characterized in that the amount of cationic retention agent is within the range of from 0.01 to 3 per cent by weight, based on dry fibres and optional fillers.

9. A process according to claim 1, 4 or 5, characterized in that the amount of anionic inorganic colloid is within the range of from 0.005 to 2 per cent by weight, based on dry fibres and optional fillers.

10. A process according to claim 1, characterized i that the weight ratio of polyaluminum compound to anioni inorganic colloid is within the range of from 0.01:1 t 3:1.

INTERNATIONAL SEARCH REPORT

International Application No PCT/SE88/00063

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC 4

D 21 H 3/00

II. FIELDS SEARCHED

Minimum Documentation Searched [7]

Classification System	Classification Symbols
IPC 4	D 21 D 3/00; D 21 H 3/00, /02, /32, /36, /38, /66, /68, /78 .../...

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [8]

SE, NO, DK, FI classes as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT [9]

Category *	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No. [13]
Y	GB, A, 2 015 614 (PRODUITS CHIMIQUES UGINE KUHLMANN) 12 September 1979 See page 1, line 8 & NL, 7901519 BE, 874178 FR, 2418297 DE, 2907354 CH, 627216	1-10
Y	WO, A1, 86/05826 (EKA NOBEL AKTIEBOLAG) 9 October 1986 See page 8, lines 1-2 & EP, 0218674 SE, 8501652 SE, 451739 JP, 63500190	1-10
Y	US, A, 4 643 801 (JOHNSON) 17 February 1987 See claims 1 and 8 & EP, 0234513 .../...	1-10

* Special categories of cited documents: [10]

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

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IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
1988-05-03	1988 -05- 2 4
International Searching Authority	**Signature of Authorized Officer** Barbro Nilsson

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

II Fields searched (cont)

US Cl 162:158,164,168,175,180,181,164.1-7, 168.1-7, 181.1-9

V. ☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE [1]

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. ☐ Claim numbers ....., because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claim numbers ....., because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claim numbers ....., because they are dependent claims and are not drafted in accordance with the second and third sentences of PCT Rule 6.4(a).

VI. ☐ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING [2]

This International Searching Authority found multiple inventions in this international application as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.

2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4. ☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

☐ The additional search fees were accompanied by applicant's protest.

☐ No protest accompanied the payment of additional search fees.

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
A	EP, A2, 0 145 686 (CARLSSON OLOF) 19 June 1985 & SE, 8306797 US, 4582627 SE, 446969 AU, 568700	1-10

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